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ANSWER 1 MARPAT COPYRIGHT 2002 ACS

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TI Electrophotographic photoconductor showing excellent electrophotographic characteristics

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SO Jpn. Kokai Tokkyo Koho, 9 pp.

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DT Patent

LA Japanese

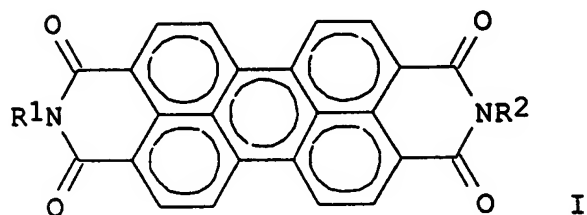
IC ICM G03G005-06

ICS G03G005-06

CC 74-3 (Radiation Chemistry, Photochemistry, and Photographic and Other Reprographic Processes)

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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PI	JP 2001154387	A2	20010608	JP 1999-332220	19991124
GI					



*acid pasting  
+ H<sub>2</sub>O/Toluene milling*

AB In the title electrophotog. photoconductor comprising a conductive support, a charge generation layer and a charge transport layer, the charge generation layer contains a mixed crystal of titanyl phthalocyanine

and perylene tetracarboxylic acid diimide represented by I (R1, R2 = H,

C1-6-alkyl, C1-16-alkoxy, C6-14-aryl, C7-20-arylalkyl). The mixed crystal

shows the x-ray diffraction max. peak at 27.2.degree..

ST electrophotog photoconductor charge generation substance mixed crystal

titanyl phthalocyanine; perylene tetracarboxylic acid diimide titanyl phthalocyanine mixed crystal electrophotog

IT Electrophotographic photoconductors (photoreceptors)

Solid solutions

(electrophotog. photoconductor contg. mixed crystal as charge

17/07/2002

generation substance for improving electrophotog. characteristics)  
IT 67075-37-0, Paliogen Black S 0084

RL: DEV (Device component use); PEP (Physical, engineering or  
chemical

process); PROC (Process); USES (Uses)  
(Paliogen Black S 0084; in charge generation mixed crystal for  
charge

generation layer of electrophotog. photoconductor showing  
excellent  
electrophotog. characteristics)

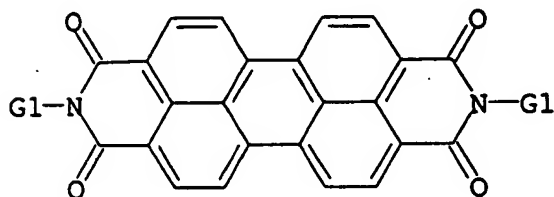
IT 26201-32-1, Titanyl phthalocyanine

RL: DEV (Device component use); PEP (Physical, engineering or  
chemical

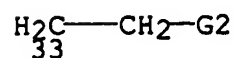
process); PROC (Process); USES (Uses)

(in charge generation mixed crystal for charge generation layer of  
electrophotog. photoconductor showing excellent electrophotog.  
characteristics)

MSTR 1



G1 = H / alkyl<(1-6)> / alkoxy<(1-6)> / aryl<(6-14)> /  
alkyl<(1-19)> (SR aryl<(-19)>) / (EX Ph (SO) / naphthyl / 33)



G2 = Ph (SO) / naphthyl  
MPL: claim 1

## PATENT ABSTRACTS OF JAPAN

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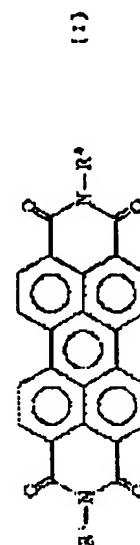
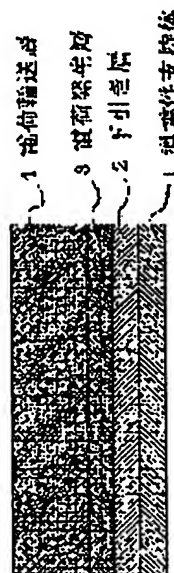
SAKIO SUSUMU

## (54) ELECTROPHOTOGRAPHIC PHOTORECEPTOR

## (57)Abstract:

**PROBLEM TO BE SOLVED:** To provide an electrophotographic photoreceptor having high sensitivity which maintains good electrostatic characteristics and which causes little double charges in the initial stage and after 10,000-cycle stress is added, and with which an image can be formed from the first rotation without requiring a process such as preliminary electrification.

**SOLUTION:** The electrophotographic photoreceptor has a charge generating layer and a charge transfer layer laminated on a conductive supporting body, and the charge generating layer contains a mixture crystal of a titanyl phthalocyanine compound and a perylene tetracarboxylic acid diimide compound expressed by general formula (I). In formula (I), each of R<sub>1</sub> and R<sub>2</sub> is independently a hydrogen atom, 1-6C alkyl group, 1-6C alkoxy group, 6-14C aryl group or 7-20C arylalkyl group.



## LEGAL STATUS

[Date of request for examination]

[Date of sending the examiner's decision of rejection]

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JP 2001-154387

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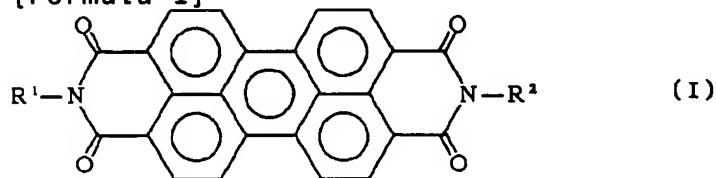
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## CLAIMS

[Claim(s)]

[Claim 1] It sets to the electrophotography photo conductor with which the laminating of a charge generating layer and the charge transporting bed was carried out on the conductive base material, and the aforementioned charge generating layer is a titanylphthalocyanine compound and the following general formula (I).

[Formula 1]

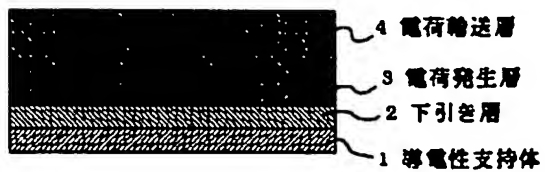


It is an electrophotography photo conductor containing a mixed-crystal object with the perylene tetrapod carboxylic-acid diimide compound expressed with (R1 and R2 show a hydrogen atom, the alkyl group of carbon numbers 1-6, the alkoxyl group of carbon numbers 1-6, the aryl group of carbon numbers 6-14, or the arylated-alkyl machine of carbon numbers 7-20 independently respectively among a formula).

[Claim 2] The electrophotography photo conductor according to claim 1 whose mixed-crystal object is what has the diffraction peak of the maximum [ Bragg angle / (2theta\*0.2 degree) / degrees / 27.2 ] in the X diffraction spectrum of CuKalpha.

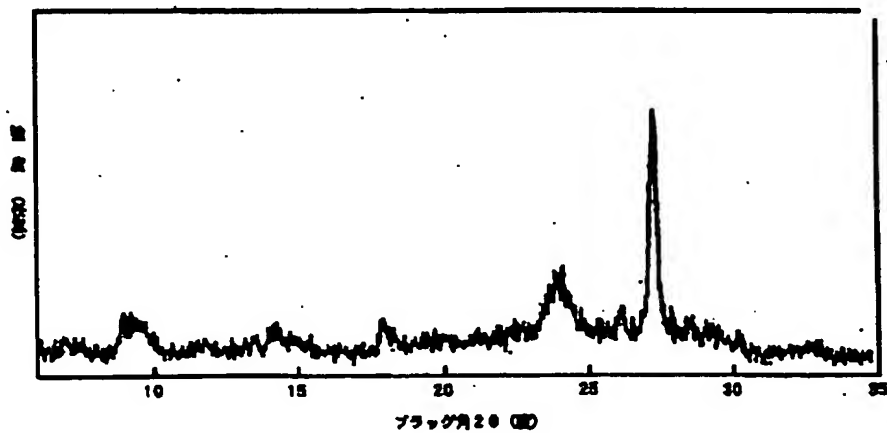
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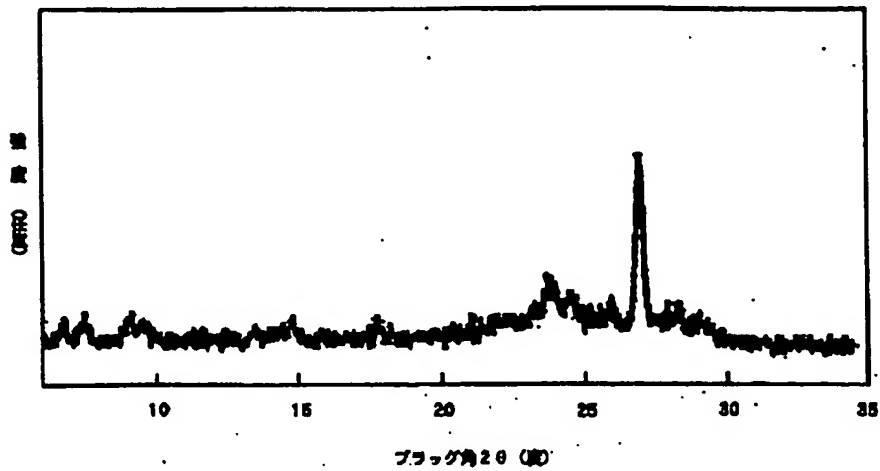


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Drawing selection [drawing]



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Drawing selection 

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Page 1 of 12

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## DETAILED DESCRIPTION

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### [Detailed Description of the Invention]

[0001]

[The technical field to which invention belongs] this invention relates to an electrophotography photo conductor.

[0002]

[Description of the Prior Art] Conventionally, inorganic photo conductive materials, such as a selenium, a zinc oxide, titanium oxide, a cadmium oxide, and an amorphous silicon, have mainly been used for the electrophotography photo conductor carried in the electrophotography equipments (a printer, copying machine, etc.) by the Carlsson method. However, in recent years, the organic photo conductive material which is excellent in respect of safety, the manufacturing cost, etc. is in use. The laminating type electrophotography photo conductor which separated the function of charge generating and charge transportation especially is produced in large quantities from the height of the sensitization by having separated the function, or material selectivity now.

[0003] Into the charge generating material of a laminating type photo conductor, it had sensitivity at near-infrared light which is semiconductor laser oscillation wavelength, such as 780nm and 660 etc. nm of Light Emitting Diode light, and it was examined broadly that composition also uses comparatively easy titanylphthalocyanine, and practical use has been presented.

[0004] An absorption spectrum and a photoconductivity not only change with kinds of central metal, but by the crystallized type, there is a difference in these physical properties and, as for titanylphthalocyanine, some examples from which the crystallized type of specification [ the titanylphthalocyanine of the same central metal ] is chosen as the photo conductors for electrophotography are reported. For example, various crystal form exists in titanylphthalocyanine and it is reported by the difference in the crystal form that there is a big difference in electrification nature, a dark decay, sensitivity, etc.

[0005] As crystal form of titanylphthalocyanine, the Bragg angle ( $2\theta^{\circ}$ ) is indicated that what gives a diffraction peak strong against 27.1 degrees is suitable 9.2 degrees, 13.1 degrees, 20.7 degrees, and 26.2 degrees by JP,59-49544,A, and the X diffraction spectrum view is shown. The vacuum evaporation film of titanylphthalocyanine is left in the saturated steam of a tetrahydrofuran for 1 to 24 hours, crystal form is changed to JP,59-166959,A, and what was used as the charge generating layer is shown. An X diffraction spectrum has few peaks, and width of face is wide, and it is shown that a Bragg angle ( $2\theta^{\circ}$ ) gives 7.5 degrees, 12.6 degrees, 13.0 degrees, 25.4 degrees, 26.2 degrees, and a diffraction peak strong against 28.6 degrees.

[0006] As crystal form of titanylphthalocyanine, the main peaks of the

Bragg angle ( $2\theta$  0.2 degree) are indicated that what it has at at least 9.5 degrees, 9.7 degrees, 11.7 degrees, 15.0 degrees, 23.5 degrees, 24.1 degrees, and 27.3 degrees is suitable by JP,64-17066,A. As crystal form of titanylphthalocyanine, the Bragg angle is indicated that what has a main diffraction peak is suitable for 27.3 degrees by JP,2-131243,A and JP,2-214867,A.

[0007] The electrophotography photo conductor using such titanylphthalocyanine as a charge generating material of a charge generating layer shows high sensitivity to near-infrared light, and although it has the outstanding property, when you make it charged with scorotron or an electrification roller, the phenomenon called double charge to which the electrification potential of 1 rotation eye becomes low compared with the electrification potential after 2 rotation eye occurs. That it is easy to produce GABURI to 1 rotation eye according to this phenomenon in reversal development, it becomes or the gradation nature of a picture changes by 1 rotation eye and 2 rotation eye. Then, after performing one or more revolution warming-up using the time which is carrying out data processing and stabilizing electrification potential, the process design which performs image formation was common.

[0008] However, since data-processing time became short sharply in connection with operation speed etc. having improved sharply in recent years, those with a commercial-scene demand that he wants to form a picture without warming-up from 1 rotation eye, and to shorten first copy speed, and the electrophotography photo conductor with little double charge are called for.

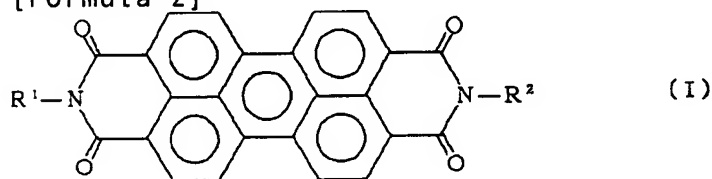
[0009]

[Problem(s) to be Solved by the Invention] Invention according to claim 1 to 2 holds a good electrostatic property, and there is little double charge after the stress of the first stage and 10,000 cycles, and it offers the high sensitivity electrophotography photo conductor which can form a picture without processes, such as preliminary electrification, from 1 rotation eye.

[0010]

[Means for Solving the Problem] For this invention, it sets to the electrophotography photo conductor with which the laminating of a charge generating layer and the charge transporting bed was carried out on the conductive base material, and the aforementioned charge generating layer is a titanylphthalocyanine compound and the following general formula (I).

[Formula 2]



It is related with the electrophotography photo conductor containing a mixed-crystal object with the perylene tetrapod carboxylic-acid diimide compound expressed with (R1 and R2 show a hydrogen atom, the alkyl group of carbon numbers 1-6, the alkoxyl group of carbon numbers 1-6, the aryl group of carbon numbers 6-14, or the arylated-alkyl machine of carbon numbers 7-20 independently respectively among a formula).

[0011] Moreover, this invention relates to the aforementioned electrophotography photo conductor which is that in which a mixed-crystal object has the diffraction peak of the maximum [ Bragg angle /

( $2\theta \approx 0.2$  degree) / degrees / 27.2 ] in the X diffraction spectrum of CuK $\alpha$ .

[0012]

[Embodiments of the Invention] Drawing 1 is the cross section showing 1 operation form of the electrophotography photo conductor concerning this invention. In drawing 1, it consists of charge transporting beds 4 applied on the under-coating layer 2 applied on the conductive base material 1 and the conductive base material 1, the charge generating layer 3 applied on the under-coating layer 2, and the charge generating layer 3.

[0013] The material which has sufficient conductivity as a conductive base material is suitable. For example, metals, such as aluminum, titanium, copper, nickel, zinc, and chromium, or the drum of these alloys, a sheet, a belt, etc. can be used. In addition, the drum which carried out electric conduction processing, a sheet, a belt, etc. can be used by carrying out the vacuum evaporation of the metallic oxides, such as metals, such as aluminum, copper, gold, silver, platinum, and palladium, and a tin oxide, indium oxide, to insulating lifters, such as plastics, glass, and paper, or distributing and applying carbon black and a metal powder to a binding resin.

[0014] Moreover, while preventing that a charge is injected into a charge generating layer from a conductive base material, in order to raise the adhesion to the conductive base material of a charge generating layer, you may prepare an under-coating layer. As an under-coating layer, the coat of well-known resins, such as a polyamide, polyvinyl alcohol, a polyvinyl butyral, polyurethane, a polyacrylic acid, a polyimide, a polycarbonate, polyester, polyethylene, and an epoxy resin, can be used. Moreover, it can form also by the resin coat which made these resins distribute the conductivity of a metal metallurgy group oxide, or a half-conductivity particle, or anodic oxide film like alumite. As thickness of an under-coating layer, 0.01-20-micrometer 0.05-5 micrometers are preferably suitable.

[0015] A charge generating layer is formed by distributing charge generating material in a binding resin. As a charge generating material, the mixed-crystal object of a perylene tetrapod carboxylic-acid diimide compound expressed with titanylphthalocyanine and the aforementioned general formula (I) is used. As a binding resin which forms a charge generating layer, a polyvinyl chloride, polyvinyl acetate, polyester, a vinyl chloride vinyl acetate copolymer, a polyvinyl butyral, a polyvinyl acetal, polystyrene, a polycarbonate, acrylic resin, phenol resin, a phenoxy resin, an epoxy resin, melamine resin, etc. are used. these resins -- independence -- or it is mixed and used As a solvent of the application liquid used in case a charge generating layer is formed with these resins, there are toluene, a methylene chloride, monochrome chlorobenzene, methyl alcohol, ethyl alcohol, ethyl acetate, a tetrahydrofuran, a cyclohexane, etc. these solvents -- independence -- or it is mixed and used

[0016] 0.05-5-micrometer about 0.1-2 micrometers are preferably suitable for the thickness of a charge generating layer. In case the application liquid which forms a charge generating layer is manufactured, as a method of distributing charge generating material in application liquid, well-known methods, such as a ball mill, a sand mill, a paint shaker, a homomixer, a homogenizer, the De Dis parser, a micronizer, and an ultrasonic wave, can be used.

[0017] The mixed-crystal object of a perylene tetrapod carboxylic-acid diimide compound expressed with the titanylphthalocyanine and the

aforementioned general formula (I) in this invention Make it precipitate underwater by the acid pay sting method, and the mixture of the perylene tetrapod carboxylic-acid diimide compound expressed with titanylphthalocyanine and the aforementioned general formula (I) is set with the X diffraction spectrum of CuKalpha. After obtaining the precipitate which has a diffraction peak characteristic of the 27.2 Bragg angles ( $2\theta \approx 0.2$  degree), what was succeedingly obtained by processing this precipitate in the aromatic system organic solvent and the mixed solvent of water is used suitably.

[0018] In addition, generally, mixture is the mere physical mixture of two or more kinds of compounds used for the raw material, and the X diffraction pattern of mixture consists of superposition of each peak pattern used for the raw material. On the other hand, the perylene tetrapod carboxylic-acid diimide compound expressed with titanylphthalocyanine and the aforementioned general formula (I) mixes the mixed-crystal object in this invention on molecule level, and an X diffraction pattern shows the pattern with which the superposition of the peak pattern of each simple substance used for the raw material differs.

[0019] When producing the mixed-crystal object of a perylene tetrapod carboxylic-acid diimide compound expressed with this titanylphthalocyanine and the aforementioned general formula (I), it is more desirable that it is 50 - 90% of the weight of a range, it is desirable that the content of the point of electrophotography properties, such as double charge, electrification nature, a rate of a dark decay, and sensitivity, to titanylphthalocyanine is 20 - 95% of the weight of a range, and it is [ 65 - 90% of the weight of especially the range is desirable, and ] most desirable that it is When there is an inclination for sensitivity to fall that the content of titanylphthalocyanine is less than 20 % of the weight and it exceeds 95 % of the weight, there is an inclination for the rate of a dark decay to fall.

[0020] It is desirable that it is that in which the point of the electrophotography properties of \*\*, such as double charge and sensitivity, to a mixed-crystal object has the diffraction peak of the maximum [ Bragg angle / ( $2\theta \approx 0.2$  degree) / degrees / 27.2 ] in the X diffraction spectrum of CuKalpha.

[0021] The above-mentioned titanylphthalocyanine can be obtained according to the publication of JP,3-71144,A, for example, can be manufactured as follows. phthalonitrile 18.4g (0.144 mols) -- alpha-chloronaphthalene 120ml -- next, 4ml (0.0364 mols) of titanium tetrachlorides is dropped [ be / under adding / it ] under nitrogen atmosphere 100-130 degrees C after carrying out a temperature up, and making it react at 200-220 degrees C after dropping for 3 hours, agitating -- the time of heat -- filtering -- alpha-chloronaphthalene -- the occasion -- a methanol washes It hydrolyzes by 140ml ion exchange water (90 degrees C, 1 hour), and a methanol washes, after repeating this operation until a solution becomes neutral. Next, it fully washes by N-methyl pyrrolidone heated at 100 degrees C, then a methanol washes. Thus, vacuum stoving of the obtained compound is carried out at 60 degrees C, and titanylphthalocyanine is obtained. (46% of yield) .

[0022] On the occasion of manufacture of the mixed-crystal object of a perylene tetrapod carboxylic-acid diimide compound expressed with above-mentioned titanylphthalocyanine and the above-mentioned aforementioned general formula (I), first, it is made to precipitate underwater by the acid pay sting method, and mixture of the perylene tetrapod carboxylic-

acid diimide compound expressed with titanylphthalocyanine and the aforementioned general formula (I) is made amorphous. Amorphous-ization by this acid pay sting method can be performed suitably as follows, for example.

[0023] First, after being preferably dropped in 40 minutes - 50 minutes for about 1 hour into 1l. of ion exchange water which cooled this by iced water after dissolving 1g of mixture of the perylene tetrapod carboxylic-acid diimide compound expressed with titanylphthalocyanine and the aforementioned general formula (I) in 50ml of concentrated sulfuric acids and agitating at a room temperature, filtration recovers precipitate. Then, it washes by ion exchange water, and subsequently, after a methanol fully washes, vacuum stoving is carried out at 60 degrees C, and amorphous powder is obtained.

[0024] Thus, in the X diffraction spectrum of the CuKalpha, other than the diffraction peak of the Bragg angle ( $2\theta \approx 0.2$  degree) clear at 27.2 degrees being shown, the peak is broad and the powder of the precipitate which consists of a perylene tetrapod carboxylic-acid diimide compound expressed with the titanylphthalocyanine and the aforementioned general formula (I) which were generated cannot specify the value clearly.

[0025] Subsequently, the mixed-crystal object of a perylene tetrapod carboxylic-acid diimide compound expressed with the above-mentioned suitable titanylphthalocyanine for this invention and the above-mentioned aforementioned general formula (I) can be acquired by changing a crystallized type by processing the powder of the precipitate (mixture of the perylene tetrapod carboxylic-acid diimide compound expressed with the titanylphthalocyanine which turned amorphously, and the aforementioned general formula (I)) obtained above in the aromatic system organic solvent and the mixed solvent of water.

[0026] As an organic solvent used at the time of crystal type conversion processed in the aromatic system organic solvent and the mixed solvent of water, benzene, toluene, a xylene, o-dichlorobenzene, etc. are mentioned, for example. At this time, as for the aromatic system organic solvent and the operating rate of water, it is desirable that the aromatic system organic solvent / water sets to 1 / 99 - 99/1 (weight ratio), and it is more desirable to be referred to as 5 / 95 - 95/5. 100 degrees C [ 40 degrees C - ] of crystal type-conversion processings in the aromatic system organic solvent and the mixed solvent of water can be preferably performed by contacting the 60-80-degree C aromatic system organic solvent and the total amount 100 weight section of the mixed solvent of water in precipitate 1 - 5 weight sections for 1 to 24 hours etc., for example.



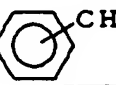

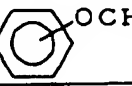



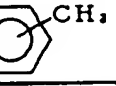

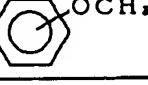






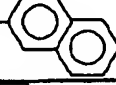

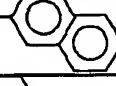
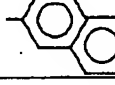


[0027] Moreover, when using as a charge generating material of an electrophotography photo conductor by performing simultaneously heating churning or pulverization, and heating churning etc. as the contact method of the aforementioned precipitate and a mixed solvent, the stable electrophotography property can be acquired. The point that the electrophotography property which heating milling processing, homogenizing, paint shaking, etc. were mentioned, and was stabilized more especially as a method of performing pulverization and heating churning simultaneously can be acquired to heating milling processing is desirable. As this diameter of a bead, being referred to as  $\phi 0.2-3\text{mm}$  is desirable, the bead [ specific gravity / of zirconia beads, an alumina bead, etc. ] used for pulverization processing of heating milling processing etc., for example, using three or more material as media is desirable, and especially the thing set to  $\phi 0.8-1.5\text{mm}$  is /

being referred to as  $\phi$ 0.5-2mm is more desirable, and / desirable.  
 [0028] For example, the processing by the concentrated sulfuric acid of the mixture of a perylene tetrapod carboxylic-acid diimide compound expressed with the previous titanylphthalocyanine and the previous aforementioned general formula (I) in a process It carries out by combination of weight ratio 0.015 less or equals of mixture/concentrated sulfuric acid. crystal type-conversion processing of subsequent precipitate (mixture of the perylene tetrapod carboxylic-acid diimide compound expressed with the titanylphthalocyanine which turned amorously, and the aforementioned general formula (I)) in the aromatic system organic solvent and the mixed solvent of water By performing pulverization and heating stirring simultaneously, it sets with the X diffraction spectrum of CuKalpha. The mixed-crystal object of a RIREN tetrapod carboxylic-acid diimide compound expressed with the titanylphthalocyanine and the aforementioned general formula (I) which have the peak of the 27.2 degree maximum of the Bragg angle ( $2\theta^{*}0.2$  degree) is acquired.

[0029] In the perylene tetrapod carboxylic-acid diimide compound expressed with the aforementioned general formula (I) used by this invention The alkyl group of the carbon numbers 1-6 which R1 and R2 in the aforementioned general formula (I) express, the alkoxyl group of carbon numbers 1-6, the aryl group of carbon numbers 6-14, or the arylated-alkyl machine of carbon numbers 7-20 You may have the substituent and the alkyl group of carbon numbers 1-4, the alkoxyl group of carbon numbers 1-4, a halogen atom, a TORIHARO alkyl group, etc. are mentioned as such a substituent. As an example, R1 and R2 in the aforementioned general formula (I) are shown and illustrated to Table 1.

[0030]

[Table 1]

化合物No.	R <sup>1</sup> 及びR <sup>2</sup>	化合物No.	R <sup>1</sup> 及びR <sup>2</sup>
A-1	$-\text{CH}_2-\text{CH}_2-$ 	A-15	$-\text{CH}_2-\text{CH}_2-$ 
A-2	$-\text{CH}_2-\text{CH}_2-$ 	A-16	$-\text{CH}_2-\text{CH}_2-$ 
A-3	$-\text{CH}_2-\text{CH}_2-$ 	A-17	
A-4	$-\text{CH}_2-$ 	A-18	
A-5	$-\text{CH}_2-$ 	A-19	
A-6	$-\text{CH}_2-$ 	A-20	
A-7	$-\text{CH}_2-\text{CH}_2-\text{CH}_3$	A-21	
A-8	$-\text{CH}_2-\text{CH}_2-\text{OCH}_3$	A-22	$-\text{CH}-$  $\text{CH}_3$
A-9	$-\text{CH}_2-\text{CH}_2-\text{CH}_2-$ 	A-23	$-\text{C}-$  $\text{CH}_3$ $\text{CH}_3$
A-10	$-\text{CH}_2-$ 	A-24	$-\text{CH}_2-\text{CH}_2-$ 
A-11	$-\text{CH}_2-$ 	A-25	$-\text{CH}_2-$ 
A-12	$-\text{H}$	A-26	
A-13	$-\text{CH}_3$	A-27	$-\text{CH}_2-\text{CH}_2-$ 
A-14	$-\text{OCH}_3$	A-28	$-\text{CH}_2-\text{CH}_2-$ 

[0031] Into the charge generating material in this invention, charge generating material other than mixed crystal with the perylene tetrapod carboxylic-acid diimide compound expressed with titanylphthalocyanine and the aforementioned general formula (I) can be used together in the range to which the property of the electrophotography photo conductor of this invention etc. does not fall if needed. As such a thing, organic pigments, such as an azoxybenzene system, a JISUAZO system, a tris azo system, a bends imidazole system, a polycyclic quinone system, an in JIGOIDO system, an anthanthrone system, a Quinacridone system, and a methine system, etc. are mentioned, for example.

[0032] The charge transporting bed made charge transportation material dissolve in a binding resin, and is formed. As a charge transportation material, with a high molecular compound, Polly N-vinylcarbazole, Halogenation Polly N-vinylcarbazole, a polyvinyl pyrene, a polyvinyl

indolo quinoxaline, Polyvinyl benzothiophene, a polyvinyl anthracene, a polyvinyl acridine, a polyvinyl pyrazoline etc. mentions -- having -- a low molecular weight compound -- full -- me -- non ON fluorene, 2, and 7-dinitro-9-full -- me -- non and 4H-indeno (1, 2, 6) thiophene-4- 3, 7-dinitro-dibenzo thiophene-5-oxide, 1-bromine pyrene, 2-phenyl pyrene, a carbazole, N-ethyl carbazole, 3-phenyl carbazole, A 3-(N-methyl-N-phenylhydrazine) methyl-9-ethyl carbazole, 2-phenylindole, 2-phenyl naphthalene, an OKISA diazole, 2, 5-screw (4-diethylaminophenyl) - 1, 3, 4-OKISA diazole, A 1-phenyl-3-(4-diethylaminostyryl)-5-(4-diethylaminostyryl)-5-(4-diethylaminophenyl) pyrazoline, A 1-phenyl-3-(p-diethylaminophenyl) pyrazoline, p -(dimethylamino)- Stilbene, 2-(4-dipropylamino phenyl)-4-(4-dimethylamino phenyl)-5-(2-chlorophenyl)-1, 3-oxazole, 2-(4-dimethylamino phenyl)-4-(4-dimethylamino phenyl)-5-(2-fluoro phenyl)-1, 3-oxazole, 2-(4-diethylaminophenyl)-4-(4-dimethylamino phenyl)-5-(2-fluoro phenyl)-1, 3-oxazole, 2-(4-dipropylamino phenyl)-4-(4-dimethylamino phenyl)-5-(2-fluoro phenyl)-1, 3-oxazole, an imidazole, a chrysene, tetrapod FEN, acridine, a triphenylamine, benzidines, these derivatives, etc. -- it is -- these well-known charges transportation material -- one sort -- or two or more sorts can be combined and it can use

[0033] As a binding resin which can be used in a charge transporting bed, silicone resin, polyamide resin, a polyurethane resin, polyester resin, an epoxy resin, poly ketone resin, polycarbonate resin, poly acrylic resin, polystyrene resin, a styrene-butadiene copolymer, a polymethyl-methacrylate resin, a polyvinyl chloride, an ethylene vinylacetate copolymer, a vinyl chloride vinyl acetate copolymer, a polyacrylamide resin, a polyvinyl carbazole, a polyvinyl pyrazoline, a polyvinyl pyrene, etc. are mentioned. Moreover, the heat-hardened type resin and the optical hardening type resin over which a bridge is constructed by heat and/or light can also be used. Anyway, it hardens by the resin, the heat, and/or light which can form a coat in the state insulating and usual, and if it is the resin which forms a coat, there will be especially no limit. these resins are independent -- or it is mixed and used

[0034] Moreover, the various additives usually used for these resins, for example, an ultraviolet ray absorbent, an antioxidant, etc., can be added suitably. In case a charge transporting bed is formed with these resins, as a solvent of application liquid, a tetrahydrofuran, a dioxane, a cyclohexane, toluene, dichloroethane, a methylene chloride, monochrome chlorobenzene, etc. can be used. these solvents are also independent -- or it can be mixed and used 5-40-micrometer about 15-30 micrometers are preferably suitable for the thickness of a charge transporting bed.

[0035] Additives, such as a plasticizer, a fluid grant agent, and a pinhole inhibitor, can be added to the application liquid for charge generating layers, or the application liquid for charge transporting beds if needed. As a plasticizer, halogenation paraffin, dimethyl naphthalene, a dibutyl phthalate, etc. are mentioned, as a fluid grant agent, MODAFURO (made in Monsanto Chemical), Acronal 4F (product made from BASUFU), etc. are mentioned, and a benzoin, dimethyl phthalate, etc. are mentioned as a pinhole inhibitor. These are used choosing them suitably and the amount should just also be determined suitably.

[0036] As a method of forming a charge generating layer or a charge transporting bed with application liquid, well-known methods, such as the spin coat method, dip coating, the roll applying method, the applicator applying method, and the wire bar applying method, are



employable. The applied charge generating layer or charge transporting bed is held and dried under heating by hot blast, infrared radiation, etc., or a vacuum.

[0037] The electrophotography photo conductor of this invention may have the protective layer on the front face from the point of abrasion resistance.

[0038]

[Example] Next, this invention is not restricted by this although this invention is explained in full detail according to an example.

[0039] The example 1 [formation of under-coating layer] alcoholic meltable polyamide resin (Japanese Lil Sun Co., Inc. make : M1276) 26.6 weight section, the melamine resin (Hitachi Chemical Co., Ltd. make : ML2000, 50 % of the weight of solid contents) 52.3 weight section, and the trimellitic anhydride (product made from Wako Pure Chem Industry) 2.8 weight section were dissolved in the ethanol 620 weight section and the 1, 1, and 2-trichloroethane 930 weight section, and application liquid was produced. By the dip painting method of construction, the obtained application liquid was applied on the cylindrical aluminum base material, was dried for 30 minutes at 140 degrees C, and the under-coating layer whose thickness is 0.3 micrometers was formed.

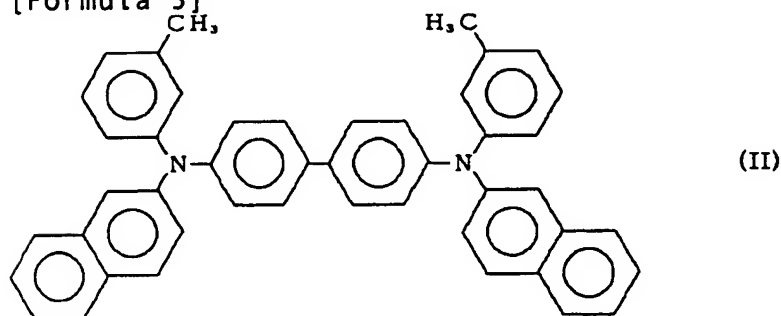
[0040] [formation of a charge generating layer] -- titanylphthalocyanine 36g and the aforementioned structure expression (A-1) N, the N'-screw (2-phenylethyl) perylene -3, 4 and 9, and 10-screw (dicarboxyimide) (the BASF A.G. make --) After dissolving in 2.4l. of sulfuric acids and agitating 48g of mixture which consists of Palliogen Black S0084 12g for 30 minutes at a room temperature, it was dropped at it, having applied it to 48l. of ion exchange water cooled by iced water for 50 minutes, and it was made to reprecipitate it. Furthermore, after agitating for 30 minutes under cooling, filtration separated precipitate. As the 1st washing, 4l. of ion exchange water was added and agitated as a wash water to precipitate, and, subsequently filtration recovered precipitate. Same washing operation was performed further 5 times in succession. Then, after 4l. of methanols washed 3 times, vacuum stoving was carried out at 60 degrees C for 4 hours, and the obtained precipitate was dried. As a result of measuring the X diffraction spectrum of the obtained dry matter, the peak of the Bragg angle ( $2\theta \approx 0.2$  degree) clear at 27.2 degrees was shown. In addition, the X diffraction spectrum was measured using RAD-IIIA made from Physical science Electrical machinery.

[0041] Subsequently, 700g [ of ion exchange water ] and toluene 250g and 1mmphi zirconia-beads 1kg are added to 10g of this dry matter. Grind and stir [ heating ] at 60-70 degrees C for 5 hours, perform filtration and centrifugal separation after cooling, and a methanol washes enough after removing a solvent. Vacuum stoving is carried out at 60 degrees C for 4 hours, and it is a titanylphthalocyanine and N, and N'-screw (2-phenylethyl) perylene. - The mixed-crystal object of 3, 4, 9, and 10-screw (dicarboxyimide) was acquired. As a result of measuring an X diffraction spectrum for the acquired mixed-crystal object, 9.6 degrees and the diffraction peak main at 27.2 degrees of the Bragg angle ( $2\theta \approx 0.2$  degree) were shown. In addition, this X diffraction spectrum was shown in drawing 2 .

[0042] Titanylphthalocyanine [ which was obtained ] and N, and N'-screw (2-phenylethyl) perylene - 3, 4, 9, the mixed-crystal object 12 weight section of 10-screw (dicarboxyimide), the polyvinyl-butyr-al-resin (Sekisui Chemical Co., Ltd. make : S REKKU BL-S) 12 weight section, the 2-ethoxyethanol 250 weight section, and the tetrahydrofuran 250 weight

section were blended, and the ball mill distributed for 20 hours. Thus, the obtained application liquid for charge generating layers was applied by dip coating on the under-coating layer of the above-mentioned aluminum base material, was dried at 120 degrees C for 1 hour, and the charge generating layer with a thickness of 0.2 micrometers was formed. [0043] [Formation of a charge transporting bed] Next, coating of the application liquid for charge transporting beds obtained by dissolving the expressing [ with the following structure expression (II) ] charge transportation material 15 weight section and the polycarbonate resin (Mitsubishi Gas Chemical Co., Inc. make : you pyrone S-3000) 15 weight section in the methylene-chloride 155 weight section was carried out by dip coating on the above-mentioned charge generating layer, it dried at 120 degrees C for 1 hour, the charge transporting bed with a thickness of 20 micrometers was formed, and the electrophotography photo conductor was obtained.

[Formula 3]



[0044] Like example 2 example 1, precipitate was dried, 140g [ of ion exchange water ] and toluene 50g was added to 2g of this dry matter, heating stirring was carried out at 60-70 degrees C for 5 hours, centrifugal separation was performed, the methanol washed enough after removing a supernatant, vacuum stoving was carried out at 60 degrees C for 4 hours, and the mixed-crystal object was acquired.

Titanylphthalocyanine [ which was obtained ] and N, and N'-screw (2-phenylethyl) perylene - As a result of measuring the X diffraction spectrum of the mixed-crystal object of 3, 4, 9, and 10-screw (dicarboxyimide), 7.5 degrees, 24.2 degrees, and the diffraction peak main at 27.3 degrees of the Bragg angle ( $2\theta \approx 0.2$  degree) were shown. In addition, this X diffraction spectrum was shown in drawing 3. The titanylphthalocyanine and N in a charge generating layer, N'-screw (2-phenylethyl) perylene - The electrophotography photo conductor was produced like the example 1 except having changed the mixed-crystal object of 3, 4, 9, and 10-screw (dicarboxyimide).

[0045] The titanylphthalocyanine and N in an example 3 charge generating layer, N'-screw (2-phenylethyl) perylene - It is a titanylphthalocyanine 31.2g and N, and N'-screw (2-phenylethyl) perylene about the mass of 3, 4, 9, and 10-screw (dicarboxyimide). - The electrophotography photo conductor was produced like the example 1 except having changed into 3, 4, 9, and 10-screw (dicarboxyimide) 16.8g.

[0046] The titanylphthalocyanine and N in an example 4 charge generating layer, N'-screw (2-phenylethyl) perylene - It is a titanylphthalocyanine 31.2g and N, and N'-screw (2-phenylethyl) perylene about the mass of 3, 4, 9, and 10-screw (dicarboxyimide). - The electrophotography photo conductor was produced like the example 2 except having changed into 3, 4, 9, and 10-screw (dicarboxyimide) 16.8g.

[0047] The titanylphthalocyanine and N in an example of comparison 1 charge generating layer, N'-screw (2-phenylethyl) perylene - The

electrophotography photo conductor was produced like the example 1 except having changed the mixed-crystal object of the mixture of 3, 4, 9, and 10-screw (dicarboxyimide) into titanylphthalocyanine.

[0048] After dissolving in 2.4l. of sulfuric acids and agitating example of comparison 2 titanylphthalocyanine 48g for 30 minutes at a room temperature, it was dropped at it, having applied it to 48l. of ion exchange water cooled by iced water for 50 minutes, and it was made to reprecipitate it. Furthermore, after agitating for 30 minutes under cooling, filtration separated precipitate. As the 1st washing, 4l. of ion exchange water was added and agitated as a wash water to precipitate, and, subsequently filtration recovered precipitate. Same washing operation was performed further 5 times in succession. Then, after 4l. of methanols washed 3 times, vacuum stoving was carried out at 60 degrees C for 4 hours, and the obtained precipitate was dried.

[0049] Subsequently, added 700g [ of ion exchange water ], and toluene 250g, and 1mmphi zirconia-beads 1kg, and it ground and stirred [ heating ] at 60-70 degrees C for 5 hours, and filtration and centrifugal separation were performed to 10g of this dry matter after cooling, the methanol washed to it enough after removing a solvent, and vacuum stoving was carried out to it at 60 degrees C for 4 hours. It is a 9 weight sections, N, and N'-screw (2-phenylethyl) perylene about the obtained titanylphthalocyanine. - 3 weight sections, the polyvinyl-butyril-resin (Sekisui Chemical Co., Ltd. make : S REKKU BL-5) 12 weight section, the 2-ethoxyethanol 250 weight section, and the tetrahydrofuran 250 weight section were blended, and the ball mill distributed 3, 4, 9, and 10-screw (dicarboxyimide) for 20 hours. Thus, the obtained application liquid for charge generating layers was applied by dip coating on the under-coating layer of the above-mentioned aluminum base material, was dried at 120 degrees C for 1 hour, and the charge generating layer with a thickness of 0.2 micrometers was formed. The electrophotography photo conductor was produced like the example 1 except having changed the charge generating layer.

[0050] [Evaluation] The difference and the electrostatic property of the amount of double charges of the obtained electrophotography photo conductor, i.e., the electrification potential of 1 rotation eye and 2 rotation eye, were measured by electrophotography characterization equipment PDT-2000 (product made from QEA). Electrification roller seal-off-approval voltage was adjusted so that electrification potential might be set to abbreviation-600V, and the amount of double charges ( $\Delta V_0 = V_{01} - V_{02}$ ) was calculated from 1 rotation eye electrification potential  $V_{01}$  after the time of the first stage, and 10,000 cycles, and 2 rotation eye electrification potential  $V_{02}$ . The electrostatic property adjusted electrification roller seal-off-approval voltage so that electrification potential might be set to abbreviation-600V, and it asked for the rest potential  $V_r$  (-V) after irradiating the light of reduction-by-half light exposure  $E_1 / 2$  (mJ/m<sup>2</sup>), and quantity of light 20 mJ/m<sup>2</sup> when exposing by charge (%) and the retention DDR 5 of 5 seconds after, and the homogeneous light with a wavelength of 780nm.

[0051] The evaluation result in examples 1-4 and the examples 1-2 of comparison is shown in Table 2. The amount of double charges is small and the photo conductor of examples 1-4 has attained the purpose of this invention. The example 1 of comparison and the example 2 of comparison have the inadequate double charge prevention effect.

[0052]

[Table 2]

表 2

	ダブルチャージ量 $\Delta V$ . (V)		電荷保持率 DDR. (%)	半減露光量 $E_{1/2}$ (mJ/m <sup>2</sup> )	残留電位 $V_r$ (-V)
	初期	1万サイクル			
実施例1	15	20	96.5	1.05	7
実施例2	8	15	96.0	1.2	10
実施例3	12	20	95.3	1.00	8
実施例4	10	15	95.0	1.10	9
比較例1	45	65	95.8	0.90	11
比較例2	35	60	95.2	1.00	10

[0053]

[Effect of the Invention] An electrophotography photo conductor according to claim 1 to 2 holds a good electrostatic property, has little double charge after the stress of the first stage and 10,000 cycles, and is the high sensitivity thing which can form a picture without processes, such as preliminary electrification, from 1 rotation eye.

[Translation done.]